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PATENT

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of : Customer Number: 20277  
 Hideaki ONO, et al. : Confirmation Number: 4949  
 Application No.: 09/893,892 : Group Art Unit: 1742  
 Filed: June 29, 2001 : Examiner: J. Sheehan

For: EXCHANGE SPRING MAGNET POWDER AND A METHOD OF PRODUCING THE SAME

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Declaration Under 37 C.F.R. § 1.132

Mail Stop Amendment  
 Commissioner for Patents  
 P.O. Box 1450  
 Alexandria, VA 22313-1450

Sir:

I, Dr. Munekatsu Shimada, declare as follows:

1. I received a doctorate of Engineering from the University of Tokyo, Department of Applied Physics.
2. My field of specialty is magnetic materials.
3. I am employed as a Senior Engineer at Technology Research Laboratory No. 1, Nissan Research Center. I have been employed by Nissan Motor Co., Ltd. for 23 years.
4. I am a coinventor of U.S. Patent Application Serial No. 09/893,892, EXCHANGE SPRING MAGNET POWDER AND A METHOD OF PRODUCING THE SAME, filed June 29, 2001.
5. This declaration supplements the declaration under 37 C.F.R. § 1.132 filed November 10, 2004.

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6. I have read and am familiar with the disclosure of the above-captioned patent application. I have read and am familiar with the disclosure of Nomura et al., U. S. Patent No. 6,261,385 (the '385 patent).

7. At my direction and under my supervision, exchange spring magnet powder was produced using the plasma irradiation method, and by the ball mill method according to the instant invention, by preparing a starting alloy having a chemical composition described in Example 3 of the '385 patent.

8. Exchange spring magnet powder was formed by:

(Step 1) Preparing a starting alloy having a chemical composition corresponding to the formula  $Nd_2Fe_{14}B_5$  (Example 3 of the '385 patent).

(Step 2) Melting the starting alloy by high-frequency induced heating, under an atmosphere of argon.

(Step 3) Quenching the melted alloy obtained by Step 2 by using the liquid quenching method with a single roller having the rolling velocity of about 10 m/sec., under an atmosphere of argon, to provide a 30 % amorphous thin ribbon.

(Step 4) Pulverizing the alloy quenched thin ribbon obtained by Step 3 to a powder with a powder size below 500  $\mu\text{m}$  under an atmosphere of argon.

(Step 5) Amorphizing 20 g of the alloy powder, obtained by Step 4, by using the plasma irradiation method as follows:

exposing the alloy powder to high frequency argon plasma to amorphousate the alloy powder from the surface direction by plasma energy.

(Step 6) Crystallizing the amorphised alloy by heat treating at 600 °C for 10 min. under vacuum.

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9. The nanocomposite magnet powder produced by this method has crystal particles with a diameter of about 100 nm. The coercive force of the nanocomposite magnet powder formed by this method is about 1.9 kOe.

10. Exchange spring magnet powder was formed by:

(Step 1) Preparing a starting alloy having a chemical composition corresponding to the formula Nd<sub>2</sub>Fe<sub>14</sub>B<sub>5</sub> (Example 3 of the '385 patent).

(Step 2) Melting the starting alloy by high-frequency induced heating, under an atmosphere of argon.

(Step 3) Quenching the melted alloy obtained by Step 2 by using the liquid quenching method with a single roller having the rolling velocity of about 10 m/sec., under an atmosphere of argon, to provide a 30 % amorphous thin ribbon.

(Step 4) Pulverizing the alloy quenched thin ribbon obtained by Step 3 to a powder with a powder size below 500 µm under an atmosphere of argon.

(Step 5) Amorphizing 20 g of the alloy powder, obtained by Step 4, by using the ball mill method as follows:

mixing the alloy powder in a stainless steel ball mill pot together with 200 g of stainless steel balls and cyclohexane as a solvent under an atmosphere of argon,

sealing the pot under an atmosphere of argon, and

milling the alloy powder for 16 hours, so as to allow ultrafine crystal particles in each crystal grain of the alloy powder to remain in an amorphous matrix.

(Step 6) Crystallizing the amorphised alloy by heat treating at 600 °C for 10 min., under vacuum, so as to allow the ultrafine particles remaining in the amorphous matrix particles to grow continuously during the crystallization process.

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11. The nanocomposite magnet powder produced by this method has crystal particles with a diameter of about 50 nm. The coercive force of the nanocomposite magnet powder formed by this method is about 4.1 kOe.

12. The exchange spring magnet powder produced by the method described in paragraph 10 has a smaller crystal particle diameter, about 50 nm, than the nanocomposite magnet powder produced by the method described in paragraph 8, about 100 nm.

13. The exchange spring magnet powder produced by the method described in paragraph 10 has a larger coercive force, about 4.1 kOe, than the nanocomposite magnet powder produced by the method described in paragraph 8, about 1.9 kOe.

14. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further, that these statements are made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of this application or any patent issuing therefrom.

Date

7/26/2005

Munakatsu Shimada  
Dr. Munakatsu Shimada